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Method of manufacturing low molecular polysaccharide and  
its oligosaccharide

저분자 다당류 및 그의 올리고당의  
제조방법

Suckhyung Cho

Kongsu Kim

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INVENTOR	(72) : 1) Suckhyung Cho KA-dong 101, 6 <sup>th</sup> , Sambu Housing, 812-1Ogwuanri, Hongsung yup, Hongsung kun, Choongchungnamdo, The Republic of Korea. 2) Kongsu Kim 793 Bunji, Kakyungdong, Hongduckku, Chungju city, Choongchungbookdo, The Republic of Korea.
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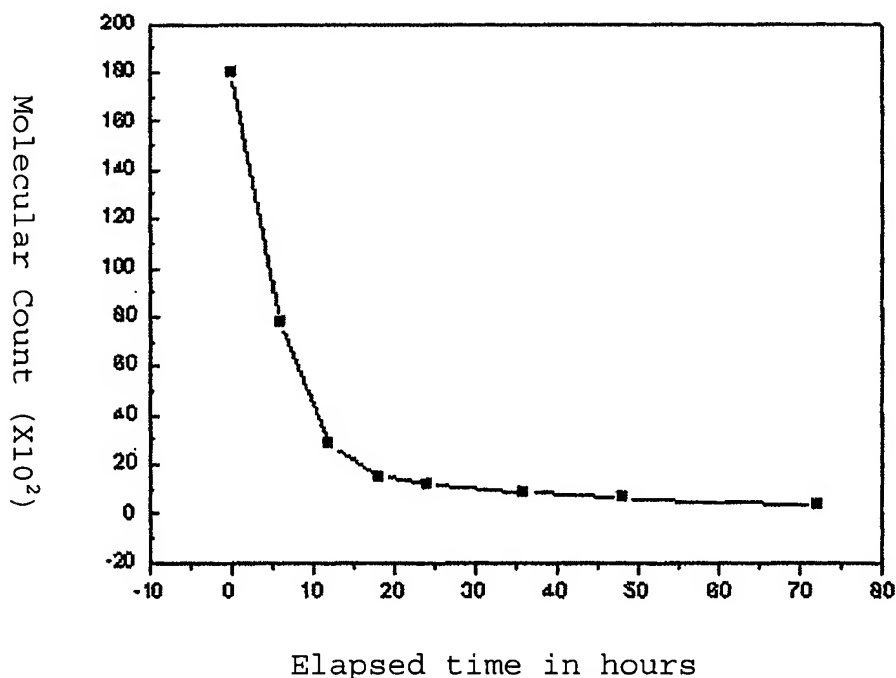
**(54) Method of manufacturing low molecular polysaccharide and  
its oligosaccharide**  
**Summary/Abstract**

In recent years, low-molecular polysaccharides and their oligosaccharide are not only expected of a broad range of applications in medical and agricultural fields, but also thought to bring a huge ripple-effect to the entire field of life sciences. Particularly, the explanation on the biological function of glycoprotein and glycolipid promises leads a way to the development of new medical products and their utilization. In plants, due to the discovery of the structure of the oligosaccharide separated from the cell walls, the defense activities of living organisms against microbic infection, and so on, they are expected to be utilized in the agriculture and the discovery of new group of materials for the functional adjustment of plants. Also, they can be used broadly in the fields of food products, cosmetics, medicines, chemical reagents, and so forth.

This invention concerns a method of manufacturing low-molecular-count polysaccharides and their oligosaccharides effectively by adding light catalysts such as titanium dioxides and peroxides in the process of dissolving polysaccharide by means of irradiating lights such as ultraviolet light, radiation and electron rays on it. These low-molecular-count polysaccharides and their oligosaccharide are bioactive substances, which can be extensively utilized in food products, natural fertilizers, medical products, etc.

Representative figures

Drawings



### Glossary terms

Light Catalyst [TN: Meaning a catalyst that performs catalytic function under Beaming/irradiation of light], Chitosan (TN: a cellulose related substance), radiation

### Details

#### Brief explanation of figures

**Fig. 1** is a graph showing the decline in molecular quantity in relation to elapsed time when a light catalyst is dispersed and Chitosan is chemically broken down, beamed with UV light at 254nm.

**Fig. 2** is a graph showing the decline in molecular quantity in relation to elapsed time when a light catalyst is dispersed and Chitosan is chemically broken down, beamed with UV light at 340nm.

**Fig. 3** is a graph showing the decline in molecular quantity in

relation to elapsed time when a light catalyst is dispersed and Chitosan is chemically broken down, beamed with gamma rays at 10 kGy.

**Fig. 4** is a graph showing the decline in molecular quantity in relation to elapsed time when a light catalyst is not dispersed and Chitosan is chemically broken down, beamed with UV light at 254nm.

**Detailed explanation of the invention.**

### **Purpose of the Invention**

### **Technologies to which the Invention Applies and the Prior Technology in that Realm**

This invention concerns a method of manufacturing low-molecular-count polysaccharides and their oligosaccharides effectively by adding light catalysts such as titanium dioxides and peroxides in the process of dissolving polysaccharide by means of irradiating lights such as ultraviolet light, radiation and electron rays on it. These low-molecular-count polysaccharides and their oligosaccharide are bioactive substances, which can be extensively utilized in food products, natural fertilizers, medical products, etc.

In recent years, low-molecular-count polysaccharides and their oligosaccharide are not only expected of a broad range of applications in medical and agricultural fields, but also thought to bring a huge ripple-effect to the entire field of life sciences. Particularly, the

explanation on the biological function of glycoprotein and glycolipid promises leads a way to the development of new medical products and their utilization. In plants, due to the discovery of the structure of the oligosaccharide separated from the cell walls, the defense activities of living organisms against microbic infection, and so on, they are expected to be utilized in the agriculture and the discovery of new group of materials for the functional adjustment of plants. Also, they can be used broadly in the fields of food products, cosmetics, medicines, chemical reagents, and so forth.

Up to the present, the method employed for deriving/obtaining low-molecular-count polysaccharide and its oligosaccharide has been to break down by fermentation or by acid.

The process of fermentation breakdown, however, owing to the unique nature of fermentation, has the drawback of limited locations where polysaccharides can be broken down and transferred, thus limiting the range of types of products obtainable. Likewise in the case of breakdown by acid, given the harsh conditions under which the breakdown of polysaccharides with acids like hydrochloric acid occurs, much monosaccharide is produced and then there exists disadvantages of not only the low conversion efficiency, but also causing environmental concerns due to the difficulty to remove the excessive quantity of chemical reagents used.

The method of manufacturing low-molecular polysaccharides by lights with complementation of these disadvantages (Patent application number 023496) (Japan Patent?10-1017040) has been presented.

However, in these pioneering preceding technologies, the majority requires long durations of time for the breakdown process, making it difficult to manufacture effectively/efficiently.

#### **In compounds that utilize light catalyst**

When a photon having energy called " $h\nu$ " is beamed on and collides with a light catalyst (TN: compound used as a catalyst), it then has energy equal to or exceeding that of the band gap of the light catalyst, and as a result as the electron comes out of the valence band and moves into the conduction band, it leaves a hole (vacant space) in the band it left. In this excited state, as it returns to the valence band, it releases energy in the form of heat. At this time, a suitable scavenger or surface bonding condition exists so that as the electron or hole becomes trapped and bonding does not occur but an oxidation-reduction reaction takes place. The valence band hole is a powerfully acidic agent and conduction band electrons become good agents for reduction. The majority of organic light-breakdown reactions directly or indirectly utilize the oxidation power of the hole. Substances that can be used as light catalysts include such materials as titanium dioxide, zinc oxide, iron oxide, cadmium sulfide, zinc sulfide, nickel oxide, cobalt oxide, etc. Titanium oxide however is known to be the best-suited material for this operation. Not only is titanium oxide, having an energy cap/gap of 3.2, is chemically and biologically stable with a low susceptibility to decay, but is very inexpensive in cost.

### **The technological objectives this invention seeks to accomplish**

This invention concerns a method of efficiently manufacturing low-molecular-count polysaccharides and their oligosaccharide by means of beaming the UV light, gamma rays, electron ray, and so on, and scattering titanium oxide zinc oxide, iron oxide, cadmium sulfide, zinc sulfide or peroxide, as a light catalyst into the powder of the polysaccharides or the solution, where polysaccharides will be dissolved by the proper solvent such as distilled water with the constant concentration as a method of manufacturing low-molecular-count polysaccharides and their oligosaccharide.

### **Structure and operation of the invention**

This invention concerns a method of manufacturing low-molecular-count polysaccharides and their oligosaccharide.

That is to say, it concerns a method of manufacturing low-molecular-count polysaccharides and their oligosaccharide efficiently, which consists of the stage where proper solvents such as distilled water and etc. dissolve polysaccharides, the stage where light catalysts such as titanium oxide zinc oxide, iron oxide, cadmium sulfide, zinc sulfide or peroxide are scattered into this solution(or, powder), and the stage where lights such as UV light, radioactive rays, and so forth are beamed.

The polysaccharides utilized by this invention are such substances as ketene, keytonic acid, pectin, Enron, gum, starch, glycogen hyaluronic



acid, polygalactosimin, their derivatives, and etc. The polysaccharide concentration used in the manufacturing process can be from 0.5 ~ 20% by weight or even in excess of 20% by weight is permissible. Also use of solids is permissible.

This invention uses, as light catalysts, titanium oxide, zinc oxide, iron oxide, cadmium sulfide, zinc sulfide, peroxide, and etc, and among them titanium oxide and hydrogen peroxide are the most suitable. The amount of light catalyst added can range between 0.0/~5% by weight but 0.5 to 1% by weight is suitable.

The light sources used by this invention include such sources UV rays and radioactive rays with UV rays being suitable in the range between 200 to 400 nm, and radioactive rays such as  $\alpha$ -ray,  $\beta$ -ray,  $\gamma$ -ray, and electron ray can be used, but among them, use of either  $\gamma$ -ray or electron ray is convenient. Intensities of electron rays 0.01kGy and 2,000kGy are used, and when used in the dried state, 100 to 2,000 kGy is used, whereas in the dissolved solution state 0.01 to 20 kGy is the desirable range.

This invention, as a means of manufacturing low-molecular-count polysaccharides and their oligosaccharides, for example, blends Chitosan 5 parts by weight, into 100~200 parts by weight of 1% acetate solution, agitating the mixture while maintaining a 5~50 degrees C to dissolve it. Then after 0.1 to 0.5 parts by weight is blended into this solution, a high-pressure mercury lamp at 254 nm irradiates it

for 30 minutes to 76 hours and Chitosan and its oligosaccharides were thus manufactured. Also, by irradiating the above Chitosan solution in radiation amounting to 0.01 to 20 kGy for 1 hour to 76 hours, low-molecular-count polysaccharides and their oligosaccharides were manufactured.

Below, a more detailed explanation is given by process execution examples.

#### **Execution example 1**

Chitosan 50 g is dissolved in 1000 grams of a 5% acetate/ water solution, and then 1 gram of titanium oxide is blended in, after which it is irradiated with a high-pressure mercury lamp at 254 nm for 1 to 76 hours manufacturing Chitosan derivatives. These derivatives were then used as they were derived either as liquid solutions, freeze dried matter (or as a dry spray). The result of this is shown in Fig. 1. As per Fig. 1, we see that, the break down/dissolution is accomplished rapidly and, as of elapsed time of twelve hours the molecular quantity declines to 2,000 and if it remains under the light illumination for more than 2 days it achieves the level of oligosaccharide. Also the quantity produced is 100% and no wastewater is produced.

#### **Execution example 2**

Except for being beamed with ultra violet light, this is executed the same as Execution example 1 above. When the UV wavelength was increased, the time required for break down increased, as seen in Fig. 2. With that exception, this example was the same as Execution

example 1.

### **Execution Example 3**

Except for being beamed with Gamma rays, this example is the same as example 1. When Gamma rays are used, as seen in Fig. 3, a low-molecular-count state is reached and oligosaccharide is produced. However, in the case of use of gamma rays, the disadvantage that occurs is that containers and facilities must be specially made for this, so higher cost is involved.

### **Execution example 4**

Except for the use of carboxymethyl cellulose or pectin, this is the same as example 1 above. When polysaccharide substitutions were made, the result was also the same.

### **Control/Comparison Example 1**

Except for the removal of Titanium Oxide, this is the same as example 1 above. As shown in Fig. 4, it can be seen that break down rate is slowed when light catalyst is not used, and productive efficiency is reduced compared to example 1.

### **Execution example 5**

The derived constituent from the break down process in each of the examples 1~3, and comparison example 1 were analyzed for their average molecule quantity by G P C, while analysis was made using callum (Shodex OHpak SB-801+SB-802+ SB803 (7.5mm L), and in the liquid derived from that, using 0.1M phosphoric acid buffering solution with the flow rate of 0.8ml/min. The result was the same as in Figures 1, 2, 3, and 4.

Results Achieved By the Invention.

As can be seen by comparison of the foregoing Execution Examples, not only a rapid rate of dissolution/break down can be achieved, but also even oligosaccharide can be dissolved in a short time by adding an irradiant catalyst. This invention is far more economical in that it can efficiently manufacture polysaccharides and oligosaccharides and produce polysaccharides with the desired molecule quantity with a low cost and in a short time by using cheap titanium oxide as a catalyst.

#### **(57) SCOPE OF CLAIMS**

##### **Claim 1**

The manufacturing method of low molecular count polysaccharides and their oligosaccharides, which consists of the step where light catalysts are scattered in a dry condition or in the solution in which polysaccharides are dissolved with the constant concentration by solvents and the step where ultraviolet light, radioactive ray, or etc. is beamed.

##### **Claim 2**

The manufacturing method of low molecular count polysaccharides and their oligosaccharides, which is characterized by the fact that light catalysts used in Claim1 such as titanium oxide, zinc oxide, iron oxide, cadmium sulfide, hydrogen peroxide, etc., can be used here, but the addition amount of the light catalysts is from 0.01 to 5% by weight.

### Claim 3

The manufacturing method of low-molecular-count polysaccharides and their oligosaccharides, which is characterized by the fact that polysaccharides substances include the ones used in Claim 1 such as pectin, ketene, Chitosan, Inulin/insulin, cellulose and derivatives, starch, glycogen, Heparin, hyaluronic acid, polygalactosimin, and their derivatives, etc.

### Claim 4

The manufacturing method of low-molecular-count polysaccharides and their oligosaccharides, which is characterized by the fact that UV light with the range of 200 to 400nm and radioactive ray from Claim1 are used and the intensities of radiation are from 0.01 to 2,000kGy.

### Figures

Figure1

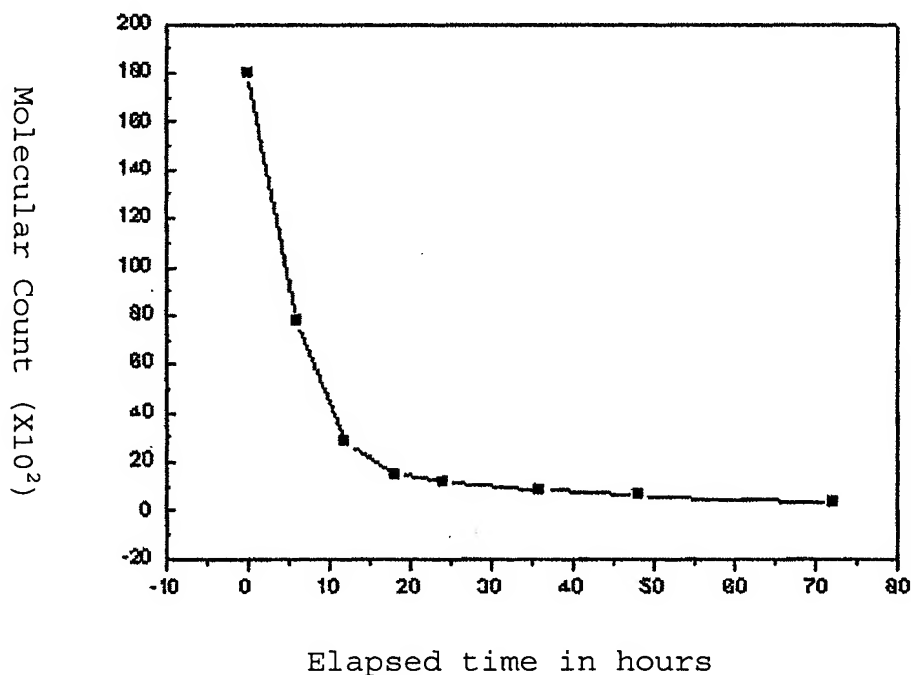


Figure2

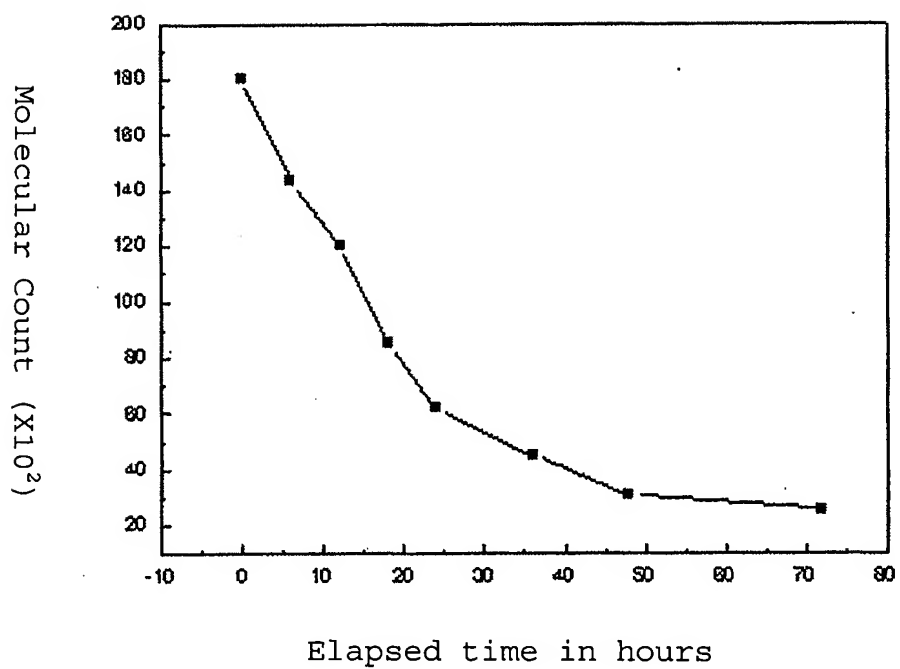


Figure3

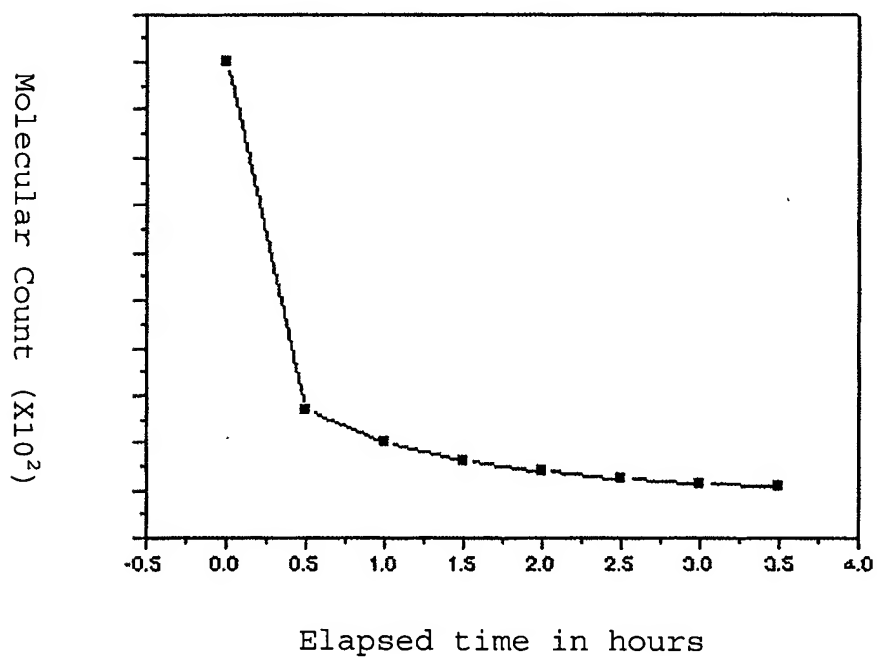


Figure4

